



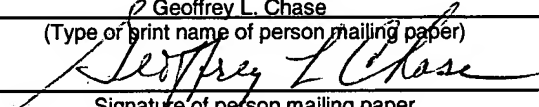
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/763,365 : Confirmation No.: 7593
Applicant : Hart, et al
Filed : January, 23, 2004
For : Process For The Purification of NF3

Art Unit : 1754
Examiner : Johnson, E.M.
Docket No. : 06408 USA
Customer No. : 23543

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

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BRIEF ON APPEAL UNDER 37 CFR 41.37(c)(1)

This appeal is from the Final Rejection of January 25, 2006.

REAL PARTY IN INTEREST

Air Products & Chemicals, Inc. is the real party in interest in the appeal. The assignment has been recorded at Reel/Frame 014931/0531.

RELATED APPEALS AND INTERFERENCES

There are no related appeals or interferences.

STATUS OF CLAIMS

Claims 1-20 are pending in the application. Claims 1-6 and 8-20 are the subject of this appeal. Claim 7 has been indicated as being allowed.

STATUS OF AMENDMENTS

No amendments have been filed subsequent to the Final Rejection of January 25, 2006.

SUMMARY OF CLAIMED SUBJECT MATTER

Claim 1 is directed to an improvement for purifying a stream containing NF_3 fluorine, hydrogen fluoride, nitrogen oxides and one that is essentially free of oxygen difluoride (Applicants' specification page 2 lines 29-31). The improvement in the purification process resides in the steps (paraphrasing), removing F_2 and HF without removing a substantial amount of NF_3 and without generating oxygen difluoride, and then removing nitrogen oxides by adsorption (Applicants' specification page 2, line 32 to page 3, line 3).

Claim 14 differentiates from Claim 1 in that the purification process includes a preamble which calls for, inter alia, the synthesis of NF_3 by reacting gaseous F_2 with ammonium acid fluoride without generating oxygen difluoride (Applicants' specification page 3, lines 15-21). The improvement in the process, e.g., the removal of HF, F_2 and nitrogen oxides resides in steps essentially identical to those in Claim 1.

Claims 1 and 14 recognize the adverse impact of generated oxygen difluoride on the life of the adsorber beds (Applicants' specification page 3, lines 22-23).

GROUND OF REJECTION TO BE REVIEWED ON APPEAL

Claims 1-6 and 8-20 stand rejected on the basis that the subject matter would have been obvious under 35 U.S.C. §103(a) over Woytek, et al., US 4,091,081 alone or in combination with Aritsuka, et al., US 4,933,158.

GROUPING OF CLAIMS

Claims 1-6 and 8-20 stand and fall together.

ARGUMENT

Examiner's Rejection of Claims 1-6, 8-9, and 12-20 Under 35 U.S.C. §103(a)

In the Final Rejection, Woytek, et al., '081 was cited as disclosing a method for forming nitrogen trifluoride comprising removal of HF and recovering nitrogen trifluoride product. The Examiner indicated that Woytek, et al., do not disclose removing fluorine and nitrogen oxides. The Examiner alleged that it would have been obvious to remove these substances, i.e., effecting removal of fluorine and nitrogen oxides from the reaction product in order to produce a purified product.

Woytek, et al., disclose passing the NF_3 containing reaction product through a KOH scrubber to remove HF and a cold trap wherein water is removed as liquid and residual trace water is removed by passing the stream through adsorbers (Woytek, et al., col. 5, lines 18-28).

At page 4, paragraph 5 of the Final Rejection in reply to Applicants' arguments in a prior amendment, the Examiner states;

“...Woytek, et al does not “generate OF₂” as Applicants appears to suggest. Rather oxygen difluoride is disclosed as a known prior art material but not in the disclosed invention of the prior art.” (See column 1, lines 25-29 of Woytek, et al. for what the Examiner appears to be referring to.)

At page 5 second paragraph of the Final Rejection, the Examiner states, “[s]ince there is no disclosure of generating OF₂ in Woytek, the claim is met.” At page 6, third paragraph, the Examiner in discussing Aritsuka, et al states, “...Aritsuka is not relied upon for a disclosure of avoiding formation of oxygen difluoride, which is rendered obvious by Woytek.”

Applicants' Response to the Rejection Under 35 U.S.C. §103(a)

It is respectfully asserted the Examiner's statements regarding the teachings of Woytek, et al., not only are incorrect, but the statements go to the heart of the present issue under 35 U.S.C. §103(a).

Woytek, et al., do in fact, although unknowingly, generate Oxygen Difluoride (OF₂) in the purification process. See another prior art reference cited of record in this case, US 4,980,144 Koto, et al., at column 1, lines 24-26, stating;

“...NF₃ gases obtained by any of the methods contain oxygen difluoride (OF₂) in most cases with a relatively large amount.”

Koto, et al., goes on to state that;

“It has been known that in the NF₃ gases prepared by the above methods, various compounds such as nitrogen (N₂), dinitrogen difluoride (N₂F₂), nitrous oxide (N₂O), carbon dioxide (CO₂) and unreacted hydrogen fluoride (HF) are contained as impurities.”

Two things are apparent from the disclosure of Koto, et al.,:

- (1) Oxygen Difluoride (OF₂) is generated in Nitrogen Trifluoride (NF₃) production and/or purification; and,

(2) Those skilled in the art did not appreciate **where** in the Nitrogen Trifluoride (NF_3) production and purification process the Oxygen Difluoride (OF_2) was emanating from, as one can see in the absence in the second quote, above, from Koto, et al., of the listing of Oxygen Difluoride (OF_2) in the list of impurities.

The significance of the Present Invention over all of the cited prior art of record is the **recognition of the Problem**, not just the **determination of the Solution** to the Problem. The Present Invention recognizes that the Problem is that Oxygen Difluoride (OF_2) is generated, not in the synthesis of the crude Nitrogen Trifluoride, **but rather in the removal of by-products of the synthesis, namely in the removal of HF and F_2 using processes, such as aqueous KOH.** See the Present Specification at page 3, paragraph [0014] and page 4 paragraph [0015]. Thus, the Present Invention claims the removal of HF and fluorine without generating Oxygen Difluoride (OF_2); namely, **the novel and unique recognition that Oxygen Difluoride (OF_2) is generated in the process of purification, and not during actual synthesis.** No one in the prior art ever recognized that Problem, before the Present Inventors recognized it and solved that Problem.

This Examiner, therefore, has misunderstood the teachings of Woytek, et al., and thus failed to understand the significance of Applicants' claim language differentiating their purification process from that described in Woytek, et al. Applicants' objection to the Examiner's rejection of Claims 1-6, 8, 9 and 12-20 lies not in whether it would have been obvious to remove impurities, such as fluorine, nitrogen oxides, etc, but in the failure to consider the steps and sequence set forth in Claims 1 and 14, particularly that:

(1) Oxygen Difluoride (OF₂) is generated during *purification*; and,

(2) *purification* needs to be adjusted or adapted to avoid the propensity of the process to generate Oxygen Difluoride (OF₂).

As stated, the Examiner's error in rejecting the claims on appeal lies in his stated position that Woytek, et al., do not generate Oxygen Difluoride (OF₂) (refer to Final Rejection, pages 4 and 6). The Examiner's position is inconsistent with the knowledge of those skilled in the art, such as Koto, et al., cited of record in this case and discussed above.

Applicants point out, at page 3, paragraph [0014], lines 24-30, of the Present Specification, that in the removal of HF and F₂ from the NF₃ reaction product by contact with KOH, Oxygen Difluoride (OF₂) is produced. The NF₃ stream of Woytek, et al., is passed through cold trap 30, and residual HF and fluorine in the NF₃ product stream is removed by contact with aqueous potassium hydroxide. Water is removed subsequently via cold trap 40 (Woytek, et al., col. 5, lines 19-25). Thus, with respect to the synthesis of NF₃ by the Woytek, et al., method of reacting F₂ with ammonium acid fluoride, **the Examiner is correct in that Oxygen Difluoride (OF₂) is not generated during synthesis.** But, in the purification step, particularly removal of F₂ and HF, Woytek, et al., unknowingly, do generate Oxygen Difluoride (OF₂), and this feature of Woytek, et al., and the other prior art of nitrogen trifluoride production purification, is excluded by Applicants' claims.

Applicants' Claims 1 and 14 call for the removal of HF and F₂ without generating Oxygen Difluoride (OF₂), whereas Woytek, et al., do generate Oxygen Difluoride (OF₂).

Absent any teaching or motivation to avoid Oxygen Difluoride (OF_2) generation in the removal of HF and F_2 from an NF_3 stream prior to removal of nitrogen oxides, the shortcomings of the teachings of the combination of prior art cited by the Examiner is fatal to maintaining a rejection of independent Claims 1 and 14, and the dependant claims therefrom under 35 U.S.C. §103(a).

At the top of page 3 of the Final Rejection in the rejection of Claims 2, 4-6 and 16-18, the Examiner noted that Woytek, et al., disclose the use of KOH in its purification process and that would at least suggest the use of Applicants' alkaline earth hydroxides, including magnesium or calcium. Applicants' assert that whether the use of KOH by Woytek, et al., is suggestive of alkaline earth hydroxides is not material. Applicants' Claims 6-8 and 16-18 call for the use of **anhydrous** hydroxides in the removal of fluorine in order to avoid formation of Oxygen Difluoride (OF_2). Woytek, et al., on the other hand, employ **aqueous** KOH. There is no disclosure in Woytek, et al., suggesting the use of **anhydrous** hydroxides to remove fluorine. Absent any disclosure on that claim distinction, the reference fails to support a rejection of those claims under 35 U.S.C. §103(a).

With regard to the rejection of Claims 3, 12, 13 and 19-20 as set forth on page 3 of the Final Rejection, Woytek, et al., does disclose various features of the synthesis of nitrogen trifluoride, e.g., the reaction of fluorine with ammonium acid fluoride, the use of adsorbers, and the removal of HF, etc. But, the Woytek, et al., disclosure is not suggestive of the Applicants' Claims 1 and 14 reciting the removal of fluorine or HF, without generating Oxygen Difluoride (OF_2) prior to the removal of the nitrogen oxides via adsorption. The Examiner is not free to ignore that claim language in an analysis under 35 U.S.C. §103(a), because the invention must be considered as a whole. All of the claimed conditions and

sequence must be suggested by Woytek, et al., to support a rejection, and Woytek, et al., do not provide that suggestion.

At page 5 of the Final Rejection, the Examiner focused upon the fact that the claims did not exclude water, and such a limitation cannot be read into the claims. Independent Claims 1 and 14 call for a series of steps and a sequence, which allow for extended life of the adsorber beds in the NF_3 process by avoiding Oxygen Difluoride (OF_2) generation. In that regard, the process steps call for the removal of HF and fluorine from an NF_3 containing stream, without generating Oxygen Difluoride (OF_2) and then removing nitrogen oxides by adsorption.

It is Applicants' position that the claims require that Oxygen Difluoride (OF_2) not be generated. If water were present, Oxygen Difluoride (OF_2) would be generated in the fluorine removal process, per paragraph [0014] of the Present Specification, and it is clear that water, and the conditions employed, would be outside of Applicants' claim language. It is the combination of water and potassium hydroxide in Woytek, et al., which does generate small amounts of Oxygen Difluoride (OF_2), again, see paragraph [0014] of the Present Specification. It is that combination of KOH and water under conditions which result in the formation of Oxygen Difluoride (OF_2) that is distinguished by the language of Claims 1 and 14. Woytek, et al., did not discuss it or address it because Woytek, et al., was not even aware of the problem, and therefore, was not in a position to address it. The Present Inventors recognized the problem and were in a position to identify a solution to the problem. (Koto, et. al., recognize Oxygen Difluoride (OF_2) in nitrogen trifluoride reaction products, but rather than avoid it, Koto, et al., merely add an additional step to remove it after it is formed, without ever recognizing where in the production/purification process Oxygen Difluoride

(OF₂) is formed or contemplating avoidance rather than Koto, et al.'s mere removal. Of course, if one doesn't recognize when or how a by-product is generated, it is nearly impossible to avoid its generation.)

Rejection of Claims 10 and 11 Under 35 U.S.C. §103(a)
Over Woytek, et al., in View of Aritsuka, et al.,

Aritsuka, et al., was cited by the Examiner as suggesting the use of zeolites for purifying NF₃, and Woytek, et al., was cited as disclosing the use of molecular sieves. It is respectfully asserted that Aritsuka, et al., adds nothing in terms of disclosure useful in an obviousness determination of Claim 1 and its dependant claims. Although Aritsuka, et al., do suggest the use of zeolites for removing nitrogen oxides from an NF₃ stream, this was acknowledged by Applicants at page 6, paragraph [0022], lines 15-23, of the Present Specification, wherein it was stated that conventional adsorbents could be used to remove nitrogen oxides, water and byproducts. Applicants' claims are directed to avoiding the generation of Oxygen Difluoride OF₂ in effecting removal of HF and fluorine, prior to removing nitrogen oxides from the NF₃ reaction product. Aritsuka, et al., provide no teaching with respect to the initial removal of F₂ from an NF₃ stream prior to removal of nitrogen oxides as called for by Claim 1 from which Claims 10 and 11 depend.

CONCLUSION

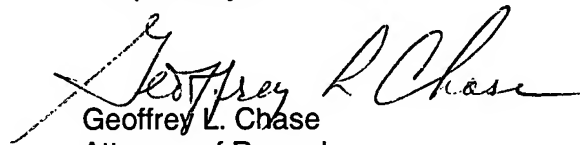
Applicants submit that independent Claims 1 and 14, and dependant Claims 2-6 and 8-20, would not have been obvious over Woytek, et al., US 4,091,081, alone, or in view of Aritsuka, et al., US 4,933,158, because these references do not establish a *prima facie* case of obviousness. The Examiner has incorrectly analyzed the Woytek, et al., NF₃ process, including purification, as one that does not generate Oxygen Difluoride OF₂ in the removal of

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residual fluorine and HF prior to removal of nitrogen oxides. Woytek, et al., provide no disclosure or motivation with respect to avoiding the generation of Oxygen Difluoride OF_2 in the purification of its NF_3 reaction product. Aritsuka, et al., offer no teaching on this point. Absent: (1) a **teaching of the problem** solved by Applicants; and, (2) a teaching of a **method of solving the problem** of Oxygen Difluoride OF_2 generation, a *prima facie* case of obviousness has not been made.

Therefore, Applicants respectfully request that the Final Rejection of Claims 1-6 and 8-20, which are the claims on appeal, under U.S.C. §103(a) be reversed, and a direction be issued to the Examiner that the application be passed to issue.

Respectfully submitted,



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CLAIMS APPENDIX

1. In a process for purifying an NF_3 stream containing fluorine, hydrogen fluoride, nitrogen oxides, and substantially free of oxygen difluoride, the improvement which comprises:

first removing hydrogen fluoride and,
removing fluorine without removing a substantial amount of NF_3 and without generating oxygen difluoride; and then,
removing the nitrogen oxides by adsorption in an adsorber, followed by recovering a purified NF_3 product.

2. The process of Claim 1 wherein the fluorine is selectively removed by contacting the stream with a metal component under conditions for forming a metal fluoride.

3. The process of Claim 1 wherein the fluorine is selectively removed by reaction with a non-metal component under conditions for forming a non-metal fluoride.

4. The process of Claim 2 in which the metal component for selectively removing fluorine is selected from the group consisting of a metal element, metal oxide, anhydrous metal hydroxide, metal sulfide, metal nitride, metal phosphide, metal arsenide, metal carbide, metal carbonate, metal silicide, metal germanide, metal boride and metal aluminide.

5. The process of Claim 4 wherein the metal element in said metal component is selected from the group consisting of tungsten, iron, zinc, silicon, aluminum, magnesium, calcium, and zirconium or mixtures thereof.

6. The process of Claim 4 wherein the fluorine is removed by contact with a metal component selected from the group consisting of aluminum oxide, anhydrous aluminum hydroxide, calcium oxide, anhydrous calcium hydroxide, strontium oxide, anhydrous strontium hydroxide, magnesium oxide, anhydrous magnesium hydroxide and lanthanum oxide.

8. The process of Claim 4 wherein the byproduct HF is removed by condensation prior to effecting removal of fluorine from the NF_3 stream.

9. The process of Claim 4 wherein HF is removed subsequent to the removal of fluorine and the HF is removed by a method selected from the group consisting of condensation, scrubbing in an aqueous alkaline hydroxide solution and adsorption.

10. The process of Claim 4 in which a zeolite is used as an adsorbent in the adsorber.

11. The process of Claim 10 in which the zeolite is selected from the group consisting of mordenite and chabazite.

12. The process of Claim 9 wherein the residual impurities in the resulting NF_3 stream from the adsorber are removed by distillation.

13. The process of Claim 3 wherein the non-metal component is selected from the group consisting of carbon and sulfur.

14. In a process for purifying NF_3 present in a reaction product stream wherein said NF_3 is produced by contacting gaseous F_2 , as the fluorine reactant, with an ammonium acid fluoride complex under conditions for generating a reaction product stream containing unreacted fluorine, byproduct hydrogen fluoride, and nitrogen oxides but substantially free of oxygen difluoride, the improvement which comprises:

first removing byproduct hydrogen fluoride, and

removing fluorine without removing a substantial amount of NF_3 and without generating oxygen difluoride; and then,

removing the nitrogen oxides by adsorption; followed by recovering a purified NF_3 product.

15. The process of Claim 14 wherein the reaction product stream contains from 5 to 20% fluorine by volume.

16. The process of Claim 15 in which a metal component is used for selectively removing fluorine and it is selected from the group consisting of a metal element, metal oxide, anhydrous metal hydroxide, metal sulfide, metal nitride, metal phosphide, metal arsenide, metal carbide, metal carbonate, metal silicide, metal germanide, metal boride and metal aluminide.

17. The process of Claim 16 wherein the metal element in said metal component is selected from the group consisting of tungsten, iron, zinc, silicon, aluminum, magnesium, calcium and zirconium or mixtures thereof.

18. The process of Claim 14 wherein the fluorine is removed by contact with a metal component selected from the group consisting of aluminum oxide, anhydrous aluminum hydroxide, calcium oxide, anhydrous calcium hydroxide, strontium oxide, anhydrous strontium hydroxide, magnesium oxide, anhydrous magnesium hydroxide and lanthanum oxide.

19. The process of Claim 14 wherein fluorine is selectively removed by reaction with a non-metal component under conditions for forming a non-metal fluoride.

20. The process of Claim 19 wherein the non-metal component is selected from the group consisting of carbon and sulfur.

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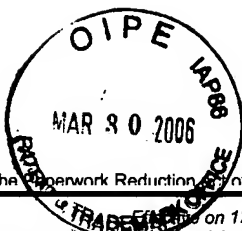
EVIDENCE APPENDIX

None

10/763,365

RELATED PROCEEDINGS APPENDIX

None



PTO/SB/17 (12-04)

Approved for use through 07/31/2006. OMB 0651-0032

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

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Effective on 12/08/2004.

FEE TRANSMITTAL

For FY 2005

☐ Applicant claims small entity status. See 37 CFR 1.27

TOTAL AMOUNT OF PAYMENT (\$) 500.00

Complete if Known

Application Number	10/763,365
Filing Date	01/23/2004
First Named Inventor	Hart, et al
Examiner Name	Johnson, E.M.
Art Unit	1754
Attorney Docket No.	06408 USA

METHOD OF PAYMENT (check all that apply)☐ Check ☐ Credit Card ☐ Money Order ☐ None ☐ Other (please identify): _____☒ Deposit Account Deposit Account Number: 01-0493 Deposit Account Name: Air Products and Chemicals, Inc.

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FEE CALCULATION**1. BASIC FILING, SEARCH, AND EXAMINATION FEES**

Application Type	FILING FEES		SEARCH FEES		EXAMINATION FEES		Fees Paid (\$)
	Fee (\$)	Small Entity Fee (\$)	Fee (\$)	Small Entity Fee (\$)	Fee (\$)	Small Entity Fee (\$)	
Utility	300	150	500	250	200	100	
Design	200	100	100	50	130	65	
Plant	200	100	300	150	160	80	
Reissue	300	150	500	250	600	300	
Provisional	200	100	0	0	0	0	

2. EXCESS CLAIM FEES

Fee Description	Fee (\$)	Small Entity Fee (\$)
Each claim over 20 or, for Reissues, each claim over 20 and more than in the original patent	50	25
Each independent claim over 3 or, for Reissues, each independent claim more than in the original patent	200	100
Multiple dependent claims	360	180

Total Claims	Extra Claims	Fee (\$)	Fee Paid (\$)	Multiple Dependent Claims	Fee (\$)	Fee Paid (\$)
_____ - 20 or HP = _____	x	50	=	0		
HP = highest number of total claims paid for, if greater than 20						
Indep. Claims	Extra Claims	Fee (\$)	Fee Paid (\$)			
_____ - 3 or HP = _____	x	200	=	0		
HP = highest number of independent claims paid for, if greater than 3						

3. APPLICATION SIZE FEE

If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$250 (\$125 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).

Total Sheets	Extra Sheets	Number of each additional 50 or fraction thereof	Fee (\$)	Fee Paid (\$)	
_____ - 100 = _____	/ 50 = _____	(round up to a whole number) x	250	=	0

4. OTHER FEE(S)

Non-English Specification, \$130 fee (no small entity discount)

Other: Appeal Brief

Fees Paid (\$)

500.00

SUBMITTED BY

Signature	Registration No. (Attorney/Agent)	Telephone
Name (Print/Type)	Date	
Geoffrey L. Chase	28,059	610-481-7265
	March 28, 2006	

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